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Microshear Bond Strength of High-viscosity Glass-ionomer to Normal and Caries-affected Dentin Under Simulated Intrapulpal Pressure

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Clinical Relevance

Current versions of high-viscosity glass-ionomer cements used with atraumatic restorative treatment show comparable bonding to normal and caries-affected dentin.

SUMMARY

Objectives: The use of high-viscosity glass-ionomer cements (HVGICs) for atraumatic restorative treatment (ART) restorations is widely practiced with the advent of various HVGICs. However, the bonding of the latter to caries-affected dentin (CAD) should be validated, especially because it is the common substrate left after conservative caries removal following the ART approach. Hence, this study was carried out to evaluate the microshear bond strength (μSBS) of three HVGICs to normal dentin (ND) and CAD under intrapulpal pressure (IPP) simulation.

Methods and Materials: The occlusal enamel of 90 molars with mid-coronal caries was cut to expose flat dentin surfaces containing both ND and CAD. Dentin substrates (ND and CAD) were differentiated using visual, tactile, caries-detecting dye, and dye-permeability methods. Prepared crown segments were equally divided (n=30) according to the tested HVGICs into GC Fuji IX GP Fast, Fuji IX GP containing chlorhexidine, and zinc-reinforced ChemFil Rock HVGIC. Microcylinders of tested HVGICs were built up on both dentin substrates (n=30 for each tested HVGIC per each substrate) using starch tubes while the specimens were subjected to simulated IPP of 15 mm Hg. The μSBS test was conducted using a universal testing machine, and failure modes were determined using a scanning electron microscope. Data were statistically analyzed using two-way analysis of variance (ANOVA) with repeated measures, one-way ANOVA, and Bonferroni *post hoc* tests (α=0.05).

Results: For both dentin substrates (ND and CAD), the μSBS values of ChemFil Rock were significantly higher than those recorded for the other HVGICs. The μSBS values of each tested HVGIC to ND and CAD were not statistically different. Failure modes were mainly mixed.

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Conclusions: Zinc-reinforced HVGIC ChemFil Rock showed superior bonding to ND and CAD compared to the GC Fuji IX GP Fast and Fuji IX with chlorhexidine. However, each of the tested HVGICs showed comparable bonding to both dentin substrates (ND and CAD).

INTRODUCTION

After more than 20 years of scientific studies, the atraumatic restorative treatment (ART) approach has become a cornerstone in global oral health.[1] ART involves the removal of soft completely demineralized carious tooth tissues with hand instruments, leaving hard, discolored caries-affected dentin (CAD), followed by the restoration of the cavity with an adhesive dental material that simultaneously seals the remaining caries-risk pits and fissures. Because of their biocompatibility, adhesive potential to tooth structure, compatibility of their coefficient of thermal expansion with that of the tooth structure, and the potential for fluoride releasing and recharging, glass-ionomer cements (GICs) are considered "smart" restorative materials to be used with ART. Despite of these outstanding advantages, GICs still suffer from shortcomings, such as early weakening of strength properties compared to resin composites, lack of command set, and moisture sensitivity.[2] Therefore, improvements were needed to overcome the above-stated drawbacks.[3]

Attempts to enhance the mechanical properties of GICs by increasing the powder/liquid ratios[4] came up with newer brands of HVGICs that were recommended for use with ART. Meta-analytic studies proved that HVGICs can be used with remarkable success in single-surface cavities; however, it fails to be routinely used in multiple-surface cavities in posterior teeth.[5-7]

Additionally, the properties of these materials can be strengthened via alteration of the glass powder composition, thus increasing the hydrolytic stability as well as the reactivity. Furthermore, the use of multicomponent fluorophosphoaluminosilicate glasses supplemented with zinc oxide particles could improve the setting reactivity and thus the material properties.[3,8]

From another aspect, researchers tried to gain a therapeutic benefit from the HVGICs through incorporating antibacterial agents, such as chlorhexidine (CHX), with glass-ionomer materials. Achieving the optimal amount of concomitant antibacterial agent without jeopardizing the basic properties of the parent materials was indeed a prime challenge. So far, various studies[9-11] have been conducted on the physical and mechanical properties of the recent types of HVGICs. Nevertheless, to date, limited studies have been focused on the bonding performance of these newer materials.

Using caries-free substrates to investigate restorative material bonding does not fully reproduce the clinical situation, where the remaining substrate after caries removal includes CAD and normal non–caries affected dentin (ND).[5] Notably, there is no published literature that deals with the bond strength of the recently introduced HVGICs to the naturally occurring CAD substrate while intrapulpal pressure (IPP) is simulated. Therefore, this study was carried out to test the μSBS of HVGICs to ND and CAD under IPP simulation.

The null hypotheses were that 1) there is no difference in the lSBS among the different HVGICs and 2) there is no difference between lSBS values of any of the tested HVGIC to both dentin substrates: ND and CAD.

METHODS AND MATERIALS

Specimen Preparation

Carious lower posterior molars were collected for the present study. From these, a total of 90 molars having occlusal caries with moderate involvement in dentin, classified according to Mount's classification as (site [1] and size [2] = 1.2) and according to ICDAS II classification (0.5), were used. Teeth were collected according to the research ethical committee guidelines.

The collected teeth were stored in phosphate-buffered saline containing 0.2% sodium azide at 4°C for a couple of weeks, during which time they were used.[12]

To expose flat dentin surfaces with ND and CAD, the occlusal enamel was trimmed perpendicularly to the long axis of each tooth using a slow-speed diamond-saw sectioning machine (Buehler Isomet, Lake Bluff, IL, USA) under water coolant. Ground dentin surfaces showing any signs of early exposure or cracks were not included in this study. To expose the pulp chamber, another cut was made parallel to the occlusal surface 2 mm apical to the cemento-enamel junction. The remnants of pulp tissue were removed from the pulp chamber using a discoid excavator (Carl Martin GmbH, Solingen, Germany) without touching its walls.[13] Each crown segment was mounted on a polymethacrylate plate containing a 19-gauge needle in the center using cyanoacrylate adhesive (Rocket Heavy, Dental Ventures of Amer-

Table 1: *Materials, Batch Numbers, Manufacturers, and Chemical Compositions of the Tested High-Viscosity Glass-Ionomer Cements*

Material/Batch Number	Manufacturer	Composition
GC Fuji IX GP Fast (Radiopaque posterior glass-ionomer restorative cement in capsules) (0804141)	GC Corp (Tokyo, Japan)	Powder 0.40 g/liquid 0.11 g (0.09 mL) per capsule
		Alumino-fluoro-silicate glass, polyacrylic acid, distilled water, polybasic carboxylic acid
Fuji IX GP containing chlorhexidine HVGIC (Radiopaque posterior glass-ionomer trial restorative cement in powder/liquid)	GC Corp	Powder: alumino-fluoro-silicate glass to which 1% chlorhexidine diacetate (Sigma Aldrich, Steinheim, Germany) was incorporated
		Liquid: polyacrylic acid, distilled water, polybasic carboxylic acid
ChemFil Rock (advanced glass-ionomer restorative material) (K79200030-03)	Dentsply De-Trey GmbH (Konstanz, Germany)	Predosed mixing capsule with a minimal dispensable amount of 0.1 mL (280 mg).
		Calcium-aluminum-zinc-fluoro-phosphor-silicate glass, polycarboxylic acid, iron oxide pigments, titanium dioxide pigments, tartaric acid, itaconic acid, water
Dentin Conditioner (GC) (280739GC)	GC Corp	20% polyacrylic acid; 3% aluminum chloride hexahydrate component

ica, Corona, CA, USA) and subsequently embedded in a chemically cured polyester resin (polyester resin #2121, Eternal Chemical Co Ltd, Hsein, Taiwan) up to the level of the cemento-enamel junction.[13]

CAD Identification

Different dentin substrates were detected with the aid of visual and tactile methods. To allow precise identification between ND and CAD, caries-detecting dye[14] and the dye permeability tests[15] were used. Red caries detector dye (Seek, Ultradent, South Jordan, UT, USA) was flooded on the flat dentin surface for 10 seconds, rinsed for five seconds, and then air-dried thoroughly for five seconds. Three different colors were revealed after drying: dark red denoting caries-infected dentin, pink color denoting CAD, and yellow color considered as ND. Partial removal of dark red–stained caries-infected dentin was carried out using a sharp spoon excavator (KLS Martin, Jacksonville, FL, USA). In the dye permeability test,[15] a 19-gauge stainless-steel butterfly needle was centrally fitted to the polymethacrylate plate and cemented to the prepared crown segment, and 10% methylene blue was permeated into the tooth through the pulp chamber under pressure. The selective staining of ND with methylene blue was attributed to the decline in the permeability of CAD compared to ND due to the presence of peritubular and intertubular crystal formation into the dentinal tubules. Therefore, the ND was stained blue, whereas the CAD was stained pale pink. The dentin surface was flattened using a rotary grinding machine and hand polished using 600-grit silicon carbide paper (MicroCut, 8 inch, Buehler) for 10 seconds to produce a standardized smear layer.[15]

Restorative Procedures

The crown segments (n=90) with ND and CAD were randomly and equally divided (n=30) according to the type of the tested HVGICs: GC Fuji IX GP Fast HVGIC (GC Corporation, Tokyo, Japan), Fuji IX GP GIC containing chlorhexidine (GC Corporation), and ChemFil Rock zinc-reinforced HVGIC (Dentsply De-Trey GmbH, Konstanz, Germany). Table 1 shows materials, batch numbers, manufacturers, and chemical compositions. All the specimens were connected to the IPP assembly under 20 mm Hg 30 minutes before and during HVGIC application. The tested HVGICs were applied according to the manufacturers' recommendations.

For the application of Fuji IX GP HVGIC (with and without chlorhexidine), the prepared dentin surface was conditioned with GC dentin conditioner (GC Corporation) for 10 seconds, rinsed thoroughly with distilled water, and blot dried. ChemFil Rock zinc-reinforced HVGIC was applied as per the manufacturer's instructions without surface conditioning. The Fuji IX GP HVGIC (containing CHX) was mixed according to the manufacturer's instructions as follows; 1 scoop (3.6 g) of powder + 1 drop (1 g) of liquid (powder/liquid ratio=1:1) were mixed manually until obtaining a homogeneous consistency. The GC Fuji IX GP Fast and ChemFil Rock capsules were activated and immediately mixed for 10 and 15 seconds, respectively, using an amalgamator (Cap-Mix, Capsule mixing device, 3M ESPE, Seefeld,

Germany). Starch tubes (pasta ZARA, Brescia, Italy) of 0.96-mm internal diameter were cut to 1-mm height to be used to build up the HVGIC microcylinders on different dentin substrates.[16]

A periodontal probe (Primadent International, Frankfurt, Germany) was used to condense the glass ionomer into the starch tubes. For each crown segment, two of the filled starch tubes were applied, one for each dentin substrate (ND and CAD) with slight finger pressure (n=30 for each tested HVGIC per each substrate). Microcylinders were coated with petroleum jelly from their top surfaces and left to set in an incubator at 37°C and a relative humidity of 100% for one hour. After the glass ionomer set, specimens were immersed in artificial saliva[17] at 37°C for four hours to soften the starch tubes. The dissolved starch tubes were carefully removed using a lancet (No. 11, Wuxi Xinda Medical Device Co, Wuxi, Jiangsu, China) leaving the HVGIC microcylinders bonded to the dentin substrates. Glass-ionomer microcylinders with interfacial gaps, bubble inclusions, or other defects were discarded and replaced. The specimens were then stored in artificial saliva at 37°C while the IPP was maintained for 24 hours in a specially constructed large incubator before testing.

Microshear Bond Strength Testing

To avoid data collection bias, blinding was considered during testing of the specimens. A specially designed attachment jig was constructed to hold the specimens to the testing machine.[15] Each specimen with its bonded glass-ionomer microcylinders was secured with four tightening bolts to the lower part of the specially designed attachment jig. The attachment jig was in turn screwed into the lower fixed and the upper movable compartments of the testing machine (Model LRX-plus, Lloyd Instruments Ltd, Ferham, UK) with a 5 KN load cell. A wire loop prepared from an orthodontic stainless-steel ligature wire 180 μm (G&H Orthodontics, Franklin, IN, USA) was wrapped around the bonded microcylinder as close as possible to its base and touching the tooth surface. A tensile load was applied at a crosshead speed of 0.5 mm/min. Data were recorded using computer software (Nexygen-MT, Lloyd Instruments).

The microshear bond strength value was calculated through dividing the load at failure by the bonding area to express the bond strength in MPa according to the following equation: $S = P/\pi r^2$, where S = microshear bond strength (in MPa), P = load at failure (in newtons), $\pi = 3.14$, and r = radius of composite microcylinder (in mm).

Statistical Analysis

Data were blindly analyzed and statistically described in terms of mean ± standard deviation. In the present study, the bond strengths to different dentin substrates were considered as the dependent variables, while the HVGICs were the independent variables. Normal distribution of the data was verified with the Kolmogorov-Smirnov test. The data were found to be normally distributed, and there was homogeneity of variance among the groups. Thus, the statistical evaluation was performed using parametric tests. Two-way analysis of variance (ANOVA) was used to determine the effect of dentin substrates and the HVGICs. It was also used to detect any significant interactions between these two variables. One-way ANOVA was employed to indicate the significant difference among the μSBS values of the tested HVGICs bonded to ND or CAD. The Bonferroni test was used for pairwise comparisons. The significance level was set at $\alpha = 0.05$. To compare the μSBS values of each HVGIC to ND and CAD, Student t-test was used. All statistical calculations were performed using SPSS (Statistical Package for the Social Sciences, SPSS Inc, Chicago, IL, USA) version 15 for Microsoft Windows.

Failure Mode Analysis

After measuring the bond strength, each fractured specimen was inspected using an environmental scanning electron microscope (Quanta 200, FEI Company, Philips, Eindhoven, The Netherlands) at 25 KV to determine its mode of failure. The failure mode was allocated into the following types:

Type I: Adhesive failure (failure at the interface between dentin and HVGIC)
Type II: Cohesive failure (failure in the HVGIC material)
Type III: Mixed failure (involving both adhesive and cohesive failures)

Representative photomicrographs of each type of failure were captured at various magnifications.

RESULTS

The mean and standard deviation for each experimental group are listed in Table 2. Two-way ANOVA showed a significant effect for the tested materials ($p<0.001$) but not for the dentin substrates ($p=0.150$) and their interactions ($p=0.757$).

Table 2: *Microshear Bond Strength μSBS Values (Mean [SD]) in MPa of the Tested High-Viscosity Glass-Ionomer Restorative Materials (HVGICs)*[a]

Dentin Substrates	HVGICs			
	GC Fuji IX GP Fast	Fuji IX GP- CHX	ChemFil Rock	*p*-Value
ND	6.59(2.8) aA (Ptf/tnt=0/30)	6.95(2.9) aA (Ptf/tnt=0/30)	10.51(3.9) aB (Ptf/tnt=0/30)	<0.0001
CAD	7.56(3.1) aA (Ptf/tnt=0/30)	7.27(3.2) aA (Ptf/tnt=0/30)	11.01(3.2) aB (Ptf/tnt=0/30)	<0.0001
p-value	0.20	0.69	0.59	

[a] *Different capital letters denote significant differences within rows, whereas different small letters denote significant differences within a column for each HVGIC. Abbreviations: CHX, chlorhexidine; ND, normal dentin; CAD, caries-affected dentin; Ptf/tnt, pretest failures/total number of tested microcylinders.*

Based on this, the first null hypothesis was rejected, while the second null hypothesis failed to be rejected.

For each dentin substrate (ND or CAD), one-way ANOVA revealed a significant difference among the three HVGIC values ($p>0.0001$). ChemFil Rock, a zinc-reinforced HVGIC, recorded significantly higher μSBS values than GC Fuji IX GP Fast and Fuji IX GP containing chlorhexidine, which were not statistically different from each other (Table 2). Despite that, each tested HVGIC type recorded higher mean μSBS values when bonded to CAD rather than those values when bonded to ND; *t*-test revealed no statistically significant differences between them (Table 2).

Regarding the failure modes, the tested HVGICs bonded to ND and CAD showed predominantly mixed failure (adhesive at dentin side/cohesive in glass ionomer). Figure 1 shows the percentages of the recorded failure modes. Representative SEM micrographs for some failure modes of tested specimens bonded to either ND or CAD are presented in Figure 2.

DISCUSSION

In the current study, the term "normal dentin" is used to describe caries-free dentin. This term has been used by many other authors[18-22] who believe that the term "sound dentin"[23-26] should indicate virgin dentin not subjected to any stimulus, not even an occlusal force, as in the case of an impacted third molar. A nomenclature that defines the type of dentin compared with CAD through discussions among the scientific community is still required.

Differentiation between normal and CAD substrates was carried out using caries-detecting dye and a dye permeability test. Despite the fact that the use of caries-detecting dye is popular,[14] its staining ability, which relies neither on chemical reaction with CAD tissue nor on bacterial staining, could lead to overexcavation. Thus, the dye permeability test, which was found to be a nondestructive, reliable method,[15] was used as an adjunctive way for differentiation between both dentin substrates.

Several studies were conducted to compare bond strength of both substrates (ND and CAD) for adhesive systems and resin composite[15,18,27-29] Nevertheless, few researchers tested bonding of HVGICs to ND and CAD.[30,31] In the current study, all tested HVGICs revealed no significant difference in their bonding to ND and CAD. Alves and others[30] reported equal bonding of Ketac Molar Easy Mix to ND and simulated CAD. Lenzi and others[31] confirmed these

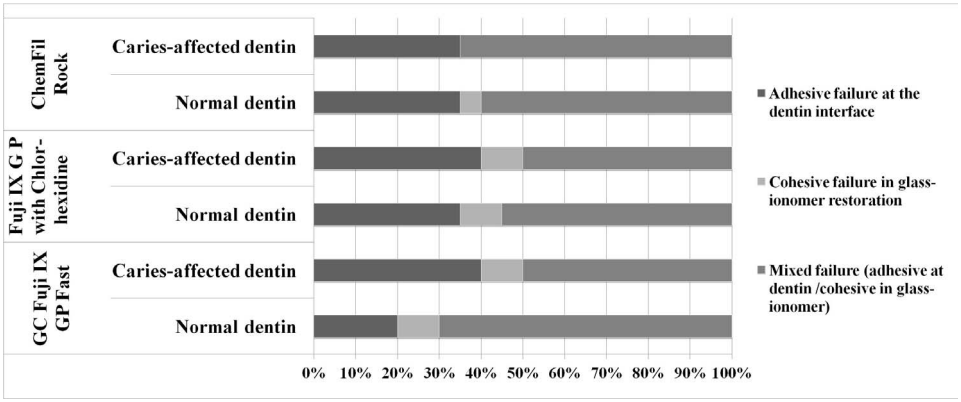

Figure 1. *The percentages of the recorded modes of failure in the tested groups.*

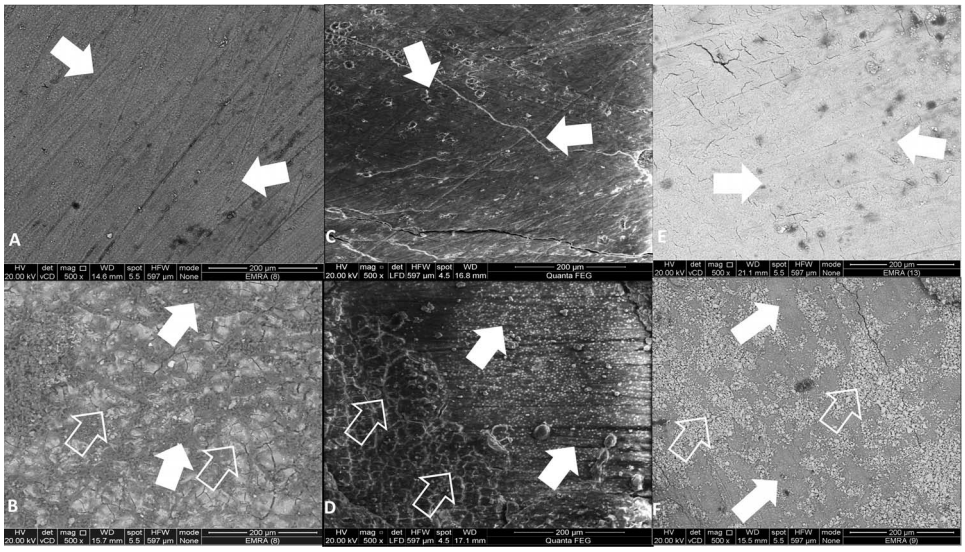

Figure 2. *SEM photomicrographs showing the predominant failure modes of GC Fuji IX GP Fast (A and B), Fuji IX GP containing chlorhexidine (C and D), and ChemFil Rock (E and F). (A, C, and E):Type I: adhesive failure (failure at the interface between dentin and GIC). (B, D, and F) Type III: mixed failure (involving both adhesive and cohesive failures). Filled arrows point to dentin, and hollow arrows point to HVGIC remnants. SEM, scanning electron micrography; GIC, glass-ionomer cement; HVGIC, high-viscosity glass-ionomer cement.*

results when they bonded Fuji IX to ND and artificially induced CAD in primary teeth.

The mechanism of GIC bonding to dentin is not precisely identified. Bonding of GICs is assumed to be a twofold mechanism, chemical interaction and micromechanical interlocking to a lesser degree. The chemical reaction is attributed to the ionic interaction between carboxylic groups from polyacids and the hydroxyapatite from the tooth surface, displacing calcium and phosphate ions from the latter. The micromechanical infiltration, which is expected to have a minor role in comparison with adhesive systems used with resin composite, may be due to the retention provided by surface irregularity of dentin and the porosity formed by the GIC self-etching property.[32]

The higher mean μSBS values obtained for all tested HVGICs bonded with CAD rather than with ND might be due to the presence of the large amount of calcium phosphate crystals occluding the dentinal tubules; these deposits favor the chemical bonding of the GICs. The histological variation between ND and CAD among carious teeth may explain why authors recorded different bond strengths of some cements to ND and CAD.

Similar to GC Fuji IX GP Fast, Fuji IX GP containing chlorhexidine HVGIC showed nonsignificant μSBS values when bonded to both dentin substrates. Previous studies showed that application of GICs to CHX-conditioned dentin surfaces has no significant effect on the bond strength.[33,34] Inclusion of 1.25% of CHX in HVGIC powder showed an antibacterial effect without causing adverse effects on mechanical properties or bond strength to ND,[10] especially when CHX diacetate was used. The tested Fuji IX contains 1% CHX diacetate,[35] which proved to be a stable material, and being a powdery component, it can be easily mixed and blended with glass-ionomer powder.[10,11] On the other hand, CHX digluconate cannot be easily separated into a powder form or even kept stable in this form. In the Fuji IX GP trial material, the concentration of CHX was kept as low as possible as was recommended. This is because CHX is not involved in the formation of the glass-ionomer matrix. A high amount of CHX would affect the network formation and the properties of the glass ionomer. This study is the first to give an idea about the bond of HVGIC containing CHX to CAD.

Generally, the setting process in Fuji IX GP HVGIC, whether containing CHX or not, like other glass-ionomer resin matrices, is developed through an acid-base reaction between the polyacid liquid and the glass powder.[36] The buildup of aluminum polyalkenoate follows the initial formation of calcium polyalkenoate. This arises in a stepwise reaction characterized mainly by an increase in strength properties over the initial 24 hours. In the reaction, precipitation of the matrix goes on until almost no ions remain in insoluble form.[37] Nevertheless, the formation of zinc-polycarboxylate complexes during the setting of ChemFil Rock GIC enhances the strength more than other complexes consisting of bivalent ions, such as calcium and strontium.[37] A new high-molecular-weight acrylic acid copolymer was also integrated in the ChemFil Rock powder, which could be another reason for the recorded increase of its strength properties[38] besides its filler modifications. In addition, the increment of itaconic

acid in the liquid of ChemFil Rock as a comonomer might play a role in its strength properties. It was reported that incorporation of itaconic acid in the conventional commercial GIC could improve the bond strength compared to compositions without copolymer.[39] Bonding under IPP simulation done in the present study might cause changes in the bonding performance of the tested materials, and this might lead to a difference in the obtained results compared with other studies. The dentin surface was conditioned with polyacrylic acid (20%) prior to the application of GC Fuji IX GP Fast and Fuji IX GP containing chlorhexidine. ChemFil Rock HVGIC was applied, as recommended by the manufacturer's instructions, without preconditioning of the dentin surface. Yet ChemFil Rock HVGIC recorded significantly higher bonding values than that of both other types of Fuji IX GP. The smear-covered dentin surface in the case of ChemFil Rock HVGIC could have reduced the dentin permeability compared to that of preconditioned dentin with the other types. This could be the reason for superior bonding of ChemFil Rock HVGIC, as the wetter surface possibly led to weaker bond for Fuji IX HVGICs.

Mode of failure analysis using SEM has been a compulsory part of bond strength studies. The predominant mode of failure in the present study was mixed, and this finding is consistent in part with Choi and others[40] despite differences in methodology. Nevertheless, a higher percentage of adhesive failure was recorded in the present study. This may be due to the specially constructed attachment used for μSBS testing that permitted the applied force to be directed at the interface. In the current study, there was no difference among the obtained failure modes of any of the tested materials with ND and CAD that supports the bond strength results.

Conclusively, the values of the μSBS of the tested brands of the HVGICs showed that these types can be recommended to restore dentin carious cavities in cases where the ART approach is applied, as their bonding to ND and CAD were comparable. While ChemFil Rock was superior in its bonding compared to the other tested HVGICs, the clinical performance of these HVGICs in stress-bearing areas still needs to be investigated.

CONCLUSIONS

Zinc-reinforced high-viscosity glass-ionomer ChemFil Rock showed superior bonding to ND and CAD compared to GC Fuji IX GP Fast and Fuji IX with chlorhexidine. However, the bonding of each of the tested HVGICs showed comparable bonding to both dentin substrates.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Faculty of Dentistry. The approval code for this study is FDCU-05082013-EM03.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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